

UNITED STATES PATENT OFFICE.

FRANCIS I. DU PONT AND ERNEST DU PONT, OF WILMINGTON, DELAWARE, ASSIGNORS
TO U. S. F. POWDER COMPANY, OF WILMINGTON, DELAWARE, A CORPORATION OF
DELAWARE.

PROCESS OF MAKING A SMOKELESS EXPLOSIVE POWDER.

No Drawing.

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This invention relates to processes of making smokeless and flashless powders; and it comprises a method of making a colloided nitrocellulose powder suitable for ordnance and of flashless, or substantially flashless, properties, wherein soluble nitrocellulose is intimately admixed with a flame propagating agent, which may be black powder or may be a nitrate, in the presence of a liquid, the liquid being one adapted to colloid or aid in colloiding the nitrocellulose; all as more fully hereinafter set forth and as claimed.

Colloided forms of soluble nitrocellulose form a well known type of smokeless powder. "Colloiding" is the technical term for treating nitrocellulose with various solvents in greater or less proportion, so that its original structure is obliterated. The amount of solvent may be enough to effect a solution or as is usually the case merely enough to plasticize. After colloiding, the solvent, or most of it, is removed; generally in such a way as to enable its recovery. Some of the solvent used remains with the powder and the properties of the final product are, to some extent, dependent upon this remaining solvent. In this country, as a rule a mixture of ether and alcohol is used for colloiding; but other solvents, such as acetone, amyl acetate, ethyl acetate and various esters of the fatty acids etc. etc. are sometimes used for special purposes. After colloiding, the powder is ordinarily formed into perforated grains; the term "grain" in this art meaning any unit of any size or shape adapted to be used in assembling a propellant charge for cannon. It is common practice to make each grain cylindrical, with longitudinal perforations, usually seven in number. In the ordinary way of manufacturing, the colloided material is forced through suitable dies which form it into perforated strings and these strings are then cut into short lengths, forming grains. The object of the perforations in the grains is to give a progressively increasing surface area for combustion and, thereby, an accelerating production of gases. In the combustion of smokeless powder, burning is proportional to the surface area including interior burning areas such as are afforded by the perforations, and the rate of development of combustion gases per time unit progressively increases until just before the powder perishes and disappears.

With this type of powder used in ordnance, as the projectile emerges from the gun, there is a brilliant flash or flame extending a considerable distance from the muzzle. This flame is apparently due to burning of unburnt materials by admixture with the oxygen of the air. In other words, it is a secondary combustion or explosion beyond the muzzle. Nitrocellulose does not contain sufficient oxygen for complete combustion.

This flash is highly objectionable from a military point of view and it is desirable to obviate it. In certain prior and copending applications, we have described and claimed methods of producing flashless powders and products of such methods. In a general way, the method whereby we have been enabled to obviate flash consists in giving the colloided powder a short of honeycomb structure with what we have called a flame propagating agent sealed in within the cells. The powder grain may be, and usually is, perforated in the ordinary way. With the cellular structure just described and with flame propagating agents in the cells, a new type of progressively increasing combustion is afforded and this may be, and usually is, superadded to the type given by the perforations. The flame propagating agent may, or may not, deliver a certain amount of oxygen to compensate for the deficiency in oxygen of the nitrocellulose. With an oxidant used to compensate for such deficiency, unburning gases are delivered at the muzzle. With other types of flame propagating agents, apparently the action is delivering gases which do not ignite, whether because of dilution or of cooling. In the actions in a gun, the work done on the projectile and

gas expansion are heat absorbing, while combustion is heat developing; these balancing against each other. In ordinary perforated powders, the maximum heat evolution is postponed in such a manner as to cause the gases leaving the muzzle to be very hot. By superadding the new type of acceleration, the condition of the gases leaving the muzzle is changed and they do not ignite; the flash is not produced.

As flame propagating agents, we have successfully used a wide variety of different materials; some oxidants and some not. Among the non-oxidants is ordinary black powder; and various mixtures of the same type, containing potassium nitrate and charcoal, with or without sulfur. Black powder, being quicker burning than colloided nitro-cellulose, as the flame reaches and opens a cell containing it, the black powder disappears, leaving a pit or cavity giving new surfaces along which combustion now takes place. The same result, flashlessness, is accomplished in another way if the included solid in the cells be one giving off oxygen when heated, such as potassium nitrate, potassium bichromate, etc. The evolved oxygen quickens the combustion locally along the interface between the nitrocellulose and the included particles. The result is the same: that of opening a new cavity or pit. In addition, the evolved oxygen tends to compensate for the oxygen deficiency of the nitrocellulose.

In an application filed June 12, 1918, under the Serial No. 239,531, we described and claimed a flashless powder. In this invention, soluble nitrocellulose and nitrate of ammonia (with or without some nitroglycerin) were intimately mixed and were colloided with ether and alcohol; some acetone being usually added. The result was an integral mass of colloided nitrocellulose containing included crystal fragments of nitrate of ammonia. The grains produced by this method have, so to speak, a honeycomb structure with nitrate of ammonia crystal fragments included within the cells.

In another application, filed January 24, 1919, as Serial No. 272,857, we described and claimed a flashless powder having the same honeycomb structure and containing a mixture of potassium and barium nitrates within the cells; and we also described a method of producing this powder wherein the nitrocellulose and the flame propagating agent were thoroughly mixed and incorporated, as by treating in a standard black powder mill in the presence of a suitable blending liquid; the blending liquid serving to prevent dusting and danger. Afterwards, the blending liquid was removed and the nitrocellulose, which now existed in mechanical mixture with the flame propagating agent, was colloided to make it integral and give the

honeycomb structure. The operation was, so to speak, a 2-stage operation with two different liquids employed; one in the mixing stage and the other in the colloidizing stage.

In another application, filed April 24, 1920, as Serial No. 376,267, we described the same method of operation in making a flashless powder. The specific flame propagating agent described and claimed in this application was black powder.

In another application, filed February 23, 1921, as Serial No. 417,229, we described and claimed the making of a flashless powder wherein the flame propagating agent was potassium bichromate and charcoal. The same 2-stage method of operation was employed in manufacture.

As the blending liquid to be used in these processes, there could be used anything not having solvent power on the nitrocellulose. We described particularly water and gasoline.

We have now discovered that a simpler, cheaper and more efficient operation may be accomplished by using as the blending liquid one which is subsequently useful in colloidizing. The liquid to be used in colloidizing, itself may be used in blending. In this stage of the operation, simple mechanical mixing only is wanted. The amount of moistening liquid required during blending is very little; merely enough to prevent dusting and incident dangers; and this amount may be, and usually is, too little to effect any substantial colloidizing of the nitrocellulose during the time required for blending, even where the blending liquid is an active solvent, such as a mixture of alcohol and ether. Its presence in no way prevents even and thorough admixture of the nitrocellulose, which is usually in the form of relatively short fibers, and the solid flame propagating agent, which is usually in the form of a rather fine powder. We may effect admixture and incorporation in a standard black powder mill. Or we may employ ordinary commercial kneading and mixing machines. The particular apparatus used is immaterial, as long as a very intimate mechanical admixture can be made.

In one embodiment of the present process we use alcohol as the blending liquid. This is not a solvent for nitrocellulose and therefore effects no colloidizing. In its presence simple mechanical mixing, which is what is here wanted, is facilitated. After the incorporation, the excess of alcohol, if any, is distilled off, and then the required amount of ether added.

In other embodiments of our invention, the liquid used in blending is the ordinary ether-alcohol mixture employed for colloidizing. If merely the amount necessary to pre-

vent substantial dusting and afford a certain amount of lubrication be employed, no material colloidizing takes place during the mixing operation under ordinary mixing conditions. More of the ether-alcohol mixture can be added later to effect colloidizing. However it is possible to secure good admixture of the nitrocellulose with the flame propagating agent in the presence of the liquid later to be used as a colloidizing solvent and have the solvent act merely as a blending agent during admixture, even where it is present in the full amount required for colloidizing, there being no such colloidizing or solution of nitrocellulose as will cement the fibers together and prevent uniform admixture. Colloidizing takes place later when the mass is subjected to pressure.

The questions of whether the term "colloidizing" is etymologically correct, and of the extent to which the solvent actually acts upon the fibers are subordinate to the practical consideration that during the mixing operation, the mass of nitrocellulose, flash eliminating compound and solvent is not in such a physical condition as to prevent the thorough mixing in and uniform distribution through the mass of the flash eliminating compound. After pressure is applied it would be practically impossible to mix any other solid material with the nitrocellulose, as it is then in the condition of a stiff rubber-like mass which will flow only when subjected to great pressure, while before pressure is applied it is scarcely different from a mass of slightly damp meal, which can be readily stirred. Naturally, much depends upon the physical characteristics of the two substances mixed.

If the mass during the kneading or mixing operation contains alcohol only, any excess may be removed by distillation and then the appropriate amount of ether added and mixed. If a small amount of a mixture of ether and alcohol has been used as the blending liquid, any deficiency can be compensated for by an addition of solvent prior to removing the mass from the kneading or mixing machine.

After admixture is perfected, the mixed materials are ordinarily in the form of a moist, but not wet, mass. The moist mass may be at once given what may be called a rough pressing operation; may be converted into blocks in any suitable machine by the use of comparatively low pressures. The object of this operation is to facilitate handling. The blocks are then transferred to a machine in which the material can be subjected to a heavy pressure, usually about 6000 pounds per square inch. By this pressure and during it, colloidizing is completed and the material is converted into an apparently homogeneous plastic mass. Actually, it is an integral cellular mass of nitrocellu-

lose containing the other ingredient in its cells. The mass is forced through suitable dies from which it emerges as strings usually with longitudinal perforations, commonly seven. These strings are cut into appropriate lengths and treated in the usual ways to remove and recover the colloidizing solvents.

As the solid flame propagating agent occurring in the honeycomb-like integral bodies of colloidized nitrocellulose produced under the present invention, there may be used any explosive having a rate of combustion materially greater than that of colloidized nitrocellulose, such as the various varieties of black powder; or any oxygen evolving solid, such as the various nitrates. The solid within the cell, being effectually sealed in against access of air, various hygroscopic nitrates and other oxygen yielding salts may be employed which are not ordinarily suitable in this class of powders. For example, sodium nitrate may be so used. However, ordinarily, we prefer potassium nitrate, barium nitrate, or a mixture of both. Strontium nitrate may be employed. Various bichromates and chromates give good results in making flashless powder. Potassium bichromate is particularly suitable. With any of these oxidants, the salt may be admixed with a little charcoal to take up its excess of oxygen. It then acts in the same manner as black powder. On the whole, nitrates and chromates give the most satisfactory results and are better than other oxygen yielding compounds, such as chlorates and perchlorates. These, however, may be used for special purposes. Manganese dioxid, alone or in admixture with other materials, is sometimes useful; as are peroxid of lead and oxid of mercury.

In using black powder, we ordinarily employ what is known as meal powder. Usually, we desire the included flame propagating agent in rather fine form; that is, we desire rather small cells in the honeycomb structure.

In a specific embodiment of the present process making a flashless powder adapted for use in 5 inch guns under service conditions, we proceed as follows:

We take 5 parts of meal black powder which is generally fine enough to pass a 100 mesh screen, and place it in a standard black powder wheel mill with 100 parts of pulped nitrocellulose which contains 23 per cent of its dry weight of alcohol. These ingredients are incorporated for a sufficient time to give uniform admixture—generally about 50 minutes. The mixture is then placed in a mixing machine and ether, containing diphenylamine in solution is added. The amount of ether used is about 43 per cent of the dry weight of the nitrocellulose, and contains sufficient diphenylamine, so that the

finished powder will contain 0.5 per cent of this material. The use of diphenylamine is not peculiar to flashless powder, as this material, in the amount mentioned, is commonly used as a stabilizing agent in all nitrocellulose powders. After the ether is added incorporation is continued until a uniform product is obtained—generally for about 50 minutes. It is understood that the periods given for incorporation are merely exemplary and are subject to wide variation, depending upon the temperature, speed, size and type of the mixers. If another colloid-
 10 ing solvent than ether-alcohol is used, as may be the case, a further adjustment of conditions becomes necessary. The vital matter is that a uniform admixture of the ingredients be obtained. The mixture is then put into a block making press and given
 20 a pressure to solidify it somewhat. It is then transferred to a hydraulic press operating under about 6000 pounds pressure from which it is forced through suitable dies to give longitudinal strings or cords having the
 25 usual seven longitudinal perforations. In making this particular powder the strings are cylindrical and about $\frac{1}{2}$ inch diameter. The strings are cut into $\frac{7}{8}$ inch lengths and are dried in the usual way so as to recover
 30 the solvent.

What we claim is:—

1. In the manufacture of a flashless, smokeless propellant powder, the process which comprises mechanically mixing and
 35 blending nitrocellulose and a flame propagating agent, blending being performed in the presence of a liquid having utility in colloid-
 ing nitrocellulose but effecting no colloid-
 40 ing during mixing, and afterwards completely colloid-
 ing the nitrocellulose.
2. In the manufacture of a flashless, smokeless propellant powder, the process which comprises mechanically mixing and
 45 blending nitrocellulose and black powder, blending being performed in the presence of a liquid having utility in colloid-
 ing nitrocellulose but effecting no substantial colloid-
 50 ing during mixing, and afterwards completely colloid-
 ing the nitrocellulose.
3. In the manufacture of a flashless, smokeless propellant powder, the process which comprises moistening nitrocellulose and a flame propagating agent with alcohol and mixing and blending the moist sub-
 55 stances, thereupon adding ether and completely colloid-
 ing the nitrocellulose.
4. In the manufacture of a flashless, smokeless propellant powder, the process which comprises moistening nitrocellulose
 60 and black powder with alcohol and mixing and blending the moist substances, there-
 upon adding ether and completely colloid-
 ing the nitrocellulose.
5. In the manufacture of a flashless,
 65 smokeless propellant powder, the process which comprises mechanically mixing and
 blending nitrocellulose and black powder in the presence of alcohol as a moistening
 and lubricating liquid, thereupon adding
 70 ether to convert the alcohol into a colloid-
 ing solvent, completely colloid-
 ing the nitrocellulose and forcing the material so obtained
 through a die press under heavy pressure,
 75 the dies being adapted to make the usual
 type of perforated grain.
6. In the manufacture of a flashless, smokeless propellant powder, the process which comprises mechanically mixing and
 blending nitrocellulose and black powder in the presence of alcohol as a moistening and
 80 lubricating liquid, thereupon adding ether to convert the alcohol into a colloid-
 ing solvent, completely colloid-
 ing the nitrocellulose, pressing the mixture to obtain blocks, and
 forcing the material so obtained through a
 85 die press under heavy pressure, the dies be-
 ing adapted to make the usual type of per-
 forated grain.
7. In the manufacture of a flashless, smokeless propellant powder, the process
 90 which comprises mechanically mixing and
 blending nitrocellulose and a flame propa-
 gating agent, in the presence of a liquid hav-
 ing utility in colloid-
 ing nitrocellulose but
 95 exercising no substantial colloid-
 ing during mixing.
8. In the manufacture of a flashless, smokeless propellant powder, the process which comprises mechanically mixing and
 blending nitrocellulose and black powder, in
 100 the presence of liquid having utility in col-
 loid-
 ing nitrocellulose but exercising no sub-
 stantial colloid-
 ing during mixing.
9. In the manufacture of a flashless, smokeless propellant powder, the process
 105 which comprises moistening uncolloid-
 ed nitrocellulose and a flame propagating agent
 with ether and alcohol and mixing and
 blending the moist substances without sub-
 110 stantially colloid-
 ing the nitrocellulose.
10. In the manufacture of a flashless, smokeless propellant powder, the process which comprises moistening uncolloid-
 ed nitrocellulose and black powder with ether and
 115 alcohol and mixing and blending the moist
 substances without substantially colloid-
 ing the nitrocellulose.
11. In the manufacture of a flashless, smokeless propellant powder, the process which comprises mechanically mixing and
 120 blending nitrocellulose and black powder in
 the presence of alcohol and ether as a mois-
 tening and lubricating liquid under condi-
 tions precluding substantial colloid-
 125 ing, thereafter completely colloid-
 ing the nitrocellulose and forcing the material so ob-
 tained through a die press under heavy pres-
 sure, the dies being adapted to make the
 usual type of perforated grain.
12. In the manufacture of flashless, 130

smokeless propellant powder the process adapted to make the usual type of perforated 10
which comprises mechanically mixing and grain and colloidizing being effected during
blending nitrocellulose and black powder in the pressing operations.
the presence of alcohol and ether as a mois- In testimony of which invention, we have
5 tening and lubricating liquid under condi- hereunto set our hands, at Wilmington, Del-
tions precluding substantial colloidizing, press- aware, on this fifth day of November, 1925.
ing the mixture to obtain blocks, and forc-
ing the material so obtained through a die
press under heavy pressure, the dies being

FRANCIS I. DU PONT.
ERNEST DU PONT.